*भारतीय मानक*
***Indian Standard***

**IS 12039 : 2022**

# स्पेंट ब्लीचिंग अर्थ — विशिष्टि

*( पहला पुनरीक्षण )*

# Spent Bleaching Earth — Specification

*( First Revision )*

ICS 71.100.40

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**December 2022** **Price Group 5**

Soaps and Other Surface Active Agents Sectional Committee, CHD 25

## FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Soaps and Other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

When dry triglyceride oils are bleached with clay/carbon, the spent earth cake produced from the filter press contains about 40 percent oil trapped in the bleaching earth. In order to minimize the losses of oil through spent earth, it is a practice in some industries to blow air/steam or other compressed gases to take out as much oil from the spent earth as possible. The oil removed is usually inferior to the bleached oil obtained during filtration. The removal of this oil also accelerates degradation of the remaining oil still held by the clay. Spent bleaching earth containing high levels of fatty matters, can be solvent extracted for the recovery of the oil. At present most of the spent bleaching earth is converted into low grade soaps, usually for utensils and dishes where the clay provides mild abrasive action. If the original oil has high iodine value, the oil present in the spent bleaching earth is likely to be oxidized and/or polymerized, thus making it less useful for soap.

This standard was originally published in 1986. This revision has been taken up in order to bring out the standard in the latest style and format of the Indian Standards. The relevant clauses have been added and the references have been updated. Also in this revision, a requirement for minimum oil content criteria has been added because the spent bleaching earth is sold on the basis of oil content and moisture content. The test method for the determination of oil content has also been added. The maximum requirement for moisture and volatile content has been modified from 5 percent to 12.5 percent.

The composition of the Committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard*

# SPENT BLEACHING EARTH — SPECIFICATION

*( First Revision )*

## 1 SCOPE

This standard prescribes the requirements and methods of sampling and test for spent bleaching earth.

## 2 REFERENCES

The Indian standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Indian standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

| *IS No* | *Title* |
|---|---|
| IS 286 : 2018 | Methods of sampling and test for soaps (*third revision*) |
| IS 548 | Methods of sampling and test for oils and fats |
| (Part 1) : 2021 | Sampling, physical and chemical tests |
| (Sec 1) : 2021 | Sampling |
| (Sec 2) : 2021 | Physical and chemical tests |
| IS 1070 : 1992 | Reagent grade water — Specification (*third revision*) |
| IS 3579 : 1966 | Methods of test for oilseeds |
| IS 11476 : 1985 | Glossary of terms relating to oils and fats |

## 3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 11476 shall apply.

## 4 REQUIREMENTS

**4.1** The material shall have iodine value and total fatty matter as agreed to between the purchaser and the supplier.

**4.2** The spent bleaching earth shall also conform to the requirements given in Table 1 when tested according to methods prescribed in col 4 of Table 1.

## 5 PACKING AND MARKING

### 5.1 Packing

The material shall be packed in suitable packages/containers as agreed to between the purchaser and the supplier.

**Table 1 Requirements for Spent Bleaching Earth**
(*Clause* 4.2)

| **Sl No.** | **Characteristic** | **Requirement** | **Method of Test, Ref To** |
|---|---|---|---|
| (1) | (2) | (3) | (4) |
| i) | Moisture and volatile matter content, percent by mass, *Max* | 12.5 | IS 286 |
| ii) | Unsaponifiable matter, percent by mass on TFM, *Max* | 5.0 | IS 548 (Part 1/Sec 2) |
| iii) | Oxidized fatty acid, percent by mass on TFM, *Max* | 5.0 | Annex A |
| iv) | Oil Content, percent by mass, *Min* | 15.0 | IS 3579 |

### 5.2 Marking

**5.2.1** The packages/containers shall be securely closed and marked legibly with the following information:

a) Name of the material;
b) Manufacturer’s name and recognized trade-mark, if any;
c) Net mass of the material;
d) Batch number or lot number in code or otherwise; and
e) Month and year of manufacture.

**5.2.2** The containers shall also, in addition, be legibly and indelibly marked with the information required under the Standards of Weights and Measures (Package Commodities) Rules, 1977.

**5.2.3** *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark

## 6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 548 (Part 1/Sec 1).

# ANNEX A

[*Table* 1, *Sl No.* iii)]

# METHODS OF TEST FOR SPENT BLEACHING EARTH

## A-l QUALITY OF REAGENTS

Unless stated otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## A-2 DETERMINATION OF OXIDIZED FATTY ACID

### A-2.0 General

A known quantity of the material is saponified with alcoholic potash and the soap formed is treated with mineral acid to release the fatty acids. The petroleum ether insoluble but ethyl ether soluble matter, which is termed as oxidized fatty acid, is determined by evaporation of the ethyl extract of the residual aqueous layer left behind after the petroleum ether extraction.

### A-2.1 Apparatus

**A-2.1.1** *Stoppered Glass Cylinder* — 250 ml capacity.

**A-2.1.2** *Separating Funnel* — 500 ml capacity.

**A-2.1.3** *Flasks* — 250 ml capacity, flat bottom; and 300 ml capacity, conical.

### A-2.2 Reagents

**A-2.2.1** *Alcoholic Potassium Hydroxide Solution* — Dissolve 50 g of potassium hydroxide in 1 litre of 95 percent ethyl alcohol.

**A-2.2.2** *Dilute Hydrochloric Acid* — 1:1 by volume.

**A-2.2.3** *Methyl Orange Indicator* — Dissolve 0.1 g of methyl orange indicator in 100 ml of water.

**A-2.2.4** *Petroleum Ether* — 60 °C/80 °C distillation range.

**A-2.2.5** *Ethyl Ether*

### A-2.3 Procedure

**A-2.3.1** Weigh accurately 3 g to 5 g of the fatty matter into a 300 ml conical flask. Add 50 ml of alcoholic potash, cover with an inverted funnel and heat on a water-bath to saponify. Agitate frequently and heat for at least 30 minutes or until saponification is complete.

**A-2.3.2** Remove the watch-glass and continue heating on a water-bath with occasional agitation to evaporate the alcohol. To avoid oxidation, do not evaporate beyond a pasty mass. If necessary, add a small amount of water when most of the alcohol has evaporated.

**A-2.3.3** Add 100 ml of distilled water and heat until the soap has completely dissolved. Wash the contents into a glass-stoppered cylinder with hot distilled water, taking care not to exceed a total volume of 130 ml in the cylinder.

**A-2.3.4** Add 3 to 5 drops of indicator and neutralize with hydrochloric acid to the pink methyl orange end point. Then add 1 ml of excess acid. Rotate the cylinder gently to mix the contents.

**A-2.3.5** Cool to at least 35 °C and add 125 ml of petroleum ether. The fatty acids need not have cleared completely before adding the ether. Stopper the cylinder, shake gently and allow to stand until the petroleum ether layer separates.

**A-2.3.6** Siphon the petroleum ether layer into a 500 ml separating funnel, making sure that as little as possible of the insoluble matter which gathers at the ether-water interface is carried over into the separating funnel. If any appreciable amount of insoluble matter does siphon over into the separating funnel, it will usually settle to the bottom and shall be drained back into the extracting cylinder. Make at least 4 more similar extractions using 25 ml to 30 ml of petroleum ether, shaking the cylinder vigorously for 30 seconds with each extraction. Extractions shall be continued until the petroleum ether layer is practically colourless.

**A-2.3.7** To the acid water remaining in the extraction cylinder add 25 ml to 30 ml of ethyl ether, stopper, shake gently and allow to stand until the ether layer separates. Siphon the ethyl ether layer through a filter paper into a tared 250 ml flat bottom flask which has been dried and cooled in a desiccator. Make at least 4 more similar extractions using 25 ml to 30 ml of ethyl ether each time, and shaking the cylinder vigorously for 30 seconds with each extraction. The last ethyl ether extract shall be practically colourless.

**A-2.3.8** Filter all extracts through the same filter paper and finally wash this filter paper thoroughly with ethyl ether to recover all the oxidized acids.

**A-2.3.9** Evaporate the ethyl ether extracts on a water-bath under a gentle stream of clean dry air. Finally, dry the oxidized fatty acids in an air oven at (105 ± 2) °C for 30 minutes. Cool in a desiccator to room temperature and weigh. Repeat until constant mass, that is, to within 0.1 percent between successive weighings, is obtained.

### A-2.4 Reporting

Report the oxidized fatty acids as a percentage of the material taken for the test.

## ANNEX B

(*Foreword*)

## COMMITTEE COMPOSITION

Soaps and Other Surface Active Agents Sectional Committee, CHD 25

| *Organization* | *Representative(s)* |
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This Indian Standard has been developed from Doc No.: CHD 25 (19107).

## Amendments Issued Since Publication

| Amend No. | Date of Issue | Text Affected |
|---|---|---|
| | | |
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| | | |

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Published by BIS, New Delhi